

PATENT SPECIFICATION (11)

1 509 680

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- (21) Application No. 39799/76 (22) Filed 24 Sept. 1976 (19)
 (31) Convention Application No. 4576/75 (32) Filed 10 Oct. 1975 in
 (33) Denmark (DK)
 (44) Complete Specification published 4 May 1978
 (51) INT. CL.² C01B 25/32
 (52) Index at acceptance
 CIA D37 G47 G47D37



(54) A PROCESS FOR PRODUCING A POWERED OR GRANULATED NON-DUSTING MINERAL COMPOSITION

- (71) We, DANSK LANDBRUGS GROV-
 VARESELSKAB AXELBORG, a Danish Com-
 pany of DK-1503 Kobenhavn V, Denmark,
 do hereby declare the invention, for which
 we pray that a patent may be granted to us,
 and the method by which it is to be per-
 formed, to be particularly described in and
 by the following statement:—
 This invention relates to a process for
 producing a powdered or granulated, non-
 flocculating mineral composition containing
 known to react a preheated aqueous suspen-
 sion of calcium carbonate with preheated
 phosphoric acid under conditions producing
 continuously a foamed material, which is
 distributed in a gas wherein the reaction
 product completes the reaction and is dried
 in freely suspended state to form a powder.
 It is the object of the present invention
 to teach a simplified procedure for non-
 dusting calcium phosphate blends, such as
 di- and monocalcium phosphate, and

ERRATA

SPECIFICATION NO 1509680

Page 1, line 2, *after* VARESELSKAB *delete* AXELBORG

Page 1, line 3, *after of insert* Axelborg

THE PATENT OFFICE
 20 June 1978

Bas 49145/8

ERRATUM

Slip No. 2

SPECIFICATION NO 1509680

Page 1, Heading (54) *for* POWERED *read* POWDERED

THE PATENT OFFICE
 12 December 1978

Bas 52589/6

- 3,391,992 it is known to prepare dicalcium
 phosphate for use as fodder by reacting
 diluted superphosphoric acid and a calcium
 salt such as calcium carbonate. This pro-
 cess requires hydrolysis of the superphos-
 phoric acid as a first step and proceeds dur-
 ing intensive development of heat.
 For Norwegian Patent No. 132,685 it is
 ate in the form of ground chalk having a
 content of water of from 15 to 20 percent
 by weight, and the phosphoric acid is pre-
 ferably an ordinary commercial phosphoric
 acid of a concentration corresponding to
 50—55 percent by weight of P₂O₅.
 The process according to the invention
 will be illustrated below by way of examples.

SEE ERRATA SLIP ATTACHED

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 NON-DUSTING MINERAL COMPOSITION

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 do hereby declare the invention, for which
 we pray that a patent may be granted to us,
 and the method by which it is to be per-
 formed, to be particularly described in and
 by the following statement:—

This invention relates to a process for
 producing a powdered or granulated, non-
 flocculating mineral composition containing
 calcium phosphates, preferably for use as
 fodder, and wherein aqueous phosphoric acid
 is reacted with calcium carbonate.

Calcium phosphates, such as dicalcium
 phosphate, monocalcium phosphate and
 mixtures thereof, are used extensively as
 components in mixed feeds or feed supple-
 ments for the feeding of domestic animals.
 The said salts can be used alone or admixed
 with other vital minerals such as various
 metal compounds, for instance salts of iron,
 zinc, manganese, copper and cobalt, and, if
 desired, vitamins and other additives.

Such mineral compositions are normally
 mixed in the fodder or drinking water in
 prescribed ratios, so that the mineral re-
 quirement of the animals will be covered.

Mineral compositions or feed supplements
 having a high content of mineral com-
 pounds are normally prepared by blending
 the dry components in the desired ratio.
 Of the drawbacks attending this procedure
 may be mentioned that the prepared mix-
 tures will normally be dust-containing which
 is unpleasant or even dangerous to both
 humans and animals, because the fine par-
 ticles may penetrate into the lungs. Besides,
 it is difficult in the conventional procedure
 to obtain an adequately uniform distribu-
 tion of all the components in the mixture.

From the specification of US Patent No.
 3,391,992 it is known to prepare dicalcium
 phosphate for use as fodder by reacting
 diluted superphosphoric acid and a calcium
 salt such as calcium carbonate. This pro-
 cess requires hydrolysis of the superphos-
 phoric acid as a first step and proceeds dur-
 ing intensive development of heat.

For Norwegian Patent No. 132,685 it is

known to react a preheated aqueous suspen-
 sion of calcium carbonate with preheated
 phosphoric acid under conditions producing
 continuously a foamed material, which is
 distributed in a gas wherein the reaction
 product completes the reaction and is dried
 in freely suspended state to form a powder.

It is the object of the present invention
 to teach a simplified procedure for non-
 dusting calcium phosphate blends, such as
 di- and monocalcium phosphate, and
 mineral blends containing these salts.

The process according to the invention is
 characterized by the mixing of aqueous phos-
 phoric acid of a concentration of at least 40
 percent by weight of P_2O_5 with a finely di-
 vided calcium carbonate having a content
 of water of from 15 to 25 percent by
 weight, whereafter other desired minerals
 and vitamins are admixed and the mixture,
 if desired, is subsequently dried. In this
 process the reaction between phosphoric
 acid and calcium carbonate is effected
 rapidly and smoothly in batches and pro-
 ceeds to completion while forming a uni-
 form reaction product. This is a result of
 the presence of a substantial content of mois-
 ture in the calcium carbonate, which en-
 sures a satisfactory and uniform distribution
 of the phosphoric acid in the reaction mix.
 The finished mixtures will be non-dusty, and
 also in cases where other mineral com-
 ponents and/or vitamins or other additives
 are added. If the process is combined
 with a subsequent drying step after all the
 supplementary components have been
 added to the reaction mass, the result will
 be a certain agglomeration whereby fine par-
 ticles present in the mass will adhere to
 form a dry non-dusting granulate or powder.

It has been found to be expedient accord-
 ing to the invention to use calcium carbon-
 ate in the form of ground chalk having a
 content of water of from 15 to 20 percent
 by weight, and the phosphoric acid is pre-
 ferably an ordinary commercial phosphoric
 acid of a concentration corresponding to
 50—55 percent by weight of P_2O_5 .

The process according to the invention
 will be illustrated below by way of examples.

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SEE INSTRUCTIONS SLIP 115 2

EXAMPLE 1

While stirring vigorously 76.1 kg powdered white chalk having a content of water of 17.0% and a content of CaCO_3 of 76.4%, corresponding to 92.0% CaCO_3 of dry components, was mixed with 76.7 kg phosphoric acid solution containing 53.8% P_2O_5 or 74.3% H_3PO_4 and 25.7% of water.

During the mixture some matter evaporated, and the result was 127.2 kg solid matter with 22.1 kg free water. The end product after drying was 105.1 kg of dry non-dusting granulate containing 5.1 kg clay and/or sand and 100 kg $\text{CaPHO}_4 \cdot 2\text{H}_2\text{O}$.

EXAMPLE 2

The procedure of Example 1 was repeated using 52.0 kg powdered white chalk and 104.7 phosphoric acid of the composition specified above. The result was 139.2 kg solid matter with 35.7 kg free water, which partly evaporates. The product was dried to a granulate weighing 103.5 kg and containing 100 kg $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$.

WHAT WE CLAIM IS:—

1. A process for producing a powdered or granulated, non-dusting mineral composition containing calcium phosphates, preferably for use as fodder, including the step of mixing aqueous phosphoric acid of a concentration of at least 40 percent by weight of

P_2O_5 with a finely divided calcium carbonate having a content of water of from 15 to 25 percent by weight to react therewith.

2. A method according to claim 1, wherein the finely divided calcium carbonate is ground chalk having a content of water of from 15 to 20 percent by weight.

3. A method according to claim 1 or 2 wherein said phosphoric acid is phosphoric acid of a concentration corresponding to 50—55 percent by weight of P_2O_5 .

4. A method according to any preceding claim including the subsequent step of admixing other minerals and/or vitamins.

5. A method according to any preceding claim including the step of drying the reaction product of the method of any one of claims 1 to 3 or the mixture prepared by the method of claim 4.

6. A product manufactured by the method of any preceding claim.

7. A method for producing powdered or granulated non-dusting mineral composition containing calcium phosphates as claimed in claim 1 substantially as herein described.

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